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December 2007

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Introduction

The advanced powder metallurgy disk alloy ME3 was designed in the HSR/EPM disk program to have extended durability at 600 to 700 °C in large disks. This was achieved by designing a disk alloy with moderately high refractory element levels optimized with rapid cooling supersolvus heat treatments to produce balanced monotonic, cyclic, and time-dependent mechanical properties. The resulting baseline alloy, processing, and supersolvus heat treatment has been demonstrated to have robust processing and manufacturing characteristics, and is projected to have extended durability capabilities (ref. 1).

The advanced disk alloy Alloy 10 was designed by Honeywell Engines and Systems through several programs to allow maximum regional engine performance at higher temperatures of 700 °C and above in smaller disks. This was achieved by using higher aluminum and titanium content for higher γ' content, combined with higher levels of refractory elements for enhanced γ' strength. This alloy was optimized with rapid cooling subsolvus heat treatments to produce maximum tensile and creep strength. However, the application of supersolvus heat treatments with rapid cooling rates to Alloy 10 has produced cracks during quenching of disks (ref. 2).

There is a long-term need for disks with higher rim temperature capabilities of 760 °C or more. This would allow higher compressor exit (T3) temperatures and allow the full utilization of advanced combustor and airfoil concepts under development. An approach being considered to meet this goal for disks consists of exploring paths which modify the processing and chemistry of ME3 and Alloy 10, to possibly improve high temperature properties while preserving rapid cooling supersolvus heat treatment capabilities. An important initial step in this effort is to understand the key variations in the grain size response versus solution heat treat temperature, as a function of composition for these alloys. At relatively low solution temperatures, the undissolved large primary γ' particles pin the grain boundaries to constrain grain growth (ref. 3) to a grain size of ASTM 11–12. However, at a relatively specific temperature, the pinning process breaks down, and grain size can increase to ASTM 6–8. This temperature approximately coincides with the solvus temperature of the γ' particles. The associated grain size transition temperature (T_g) is an important parameter that dictates the heat treatment temperatures of an alloy. It sets the subsolvus (ASTM 11–12) and supersolvus (ASTM 6–8) heat treatment temperature ranges, and can influence the propensities for quench cracking and thermally induced porosity produced during the supersolvus heat treatment step (ref. 4).

The objective of this study was to determine and compare the grain size-temperature response of a series of experimental alloys with compositions around and spanning two disk superalloys, ME3 and Alloy 10. Coupons of extruded material from each alloy were soaked at various temperatures for 1 hr. They were then metallographically prepared and evaluated for grain size response. The responses were compared and related to the chemistries of these alloys.

Materials and Procedure

The chemistries in measured weight percent of the five experimental ME3 alloys identified by extrusion numbers and six experimental Alloy 10 alloys identified by extrusion letters are given in table 1 along with the base alloy chemistries. Powder of the Navy alloy NF3 was also provided courtesy of the Navy (ref. 5) from a production-scale atomization run at Homogeneous Metals. The five experimental ME3 alloys were atomized in the pilot-scale atomizer at Special Metals Corp. while the six experimental Alloy 10 compositions and baseline composition were atomized in the pilot-scale atomizer at Homogenous Metals, Inc. All powder was screened to –270 mesh, then canned, hot compacted and extruded at Wyman-Gordon Forgings. A coupon of base ME3 was obtained from a scaled-up forging previously heat treated to 1135 °C. Specimens approximately 12 mm square and 20 mm long were prepared near the leading end of each extrusion.

Specimens of each alloy were tied into bundles and heated treated in resistance heating furnaces. For each soaking temperature, the furnace containing a bundle of alloy specimens was ramped up to temperature in 3 hr then held at temperature for 1 hr. The furnace was then turned off and the specimens were slow cooled in the furnace. Specimens were sectioned in half, metallographically prepared, and swab etched with Kallings reagent. Grain size was measured according to ASTM E–112 using linear intercept procedures with circular grid overlays, while γ′ content was rated for each specimen. Statistical analyses were performed using RS/Client software. Chemistry variables were evaluated in weight percent, and were orthogonally scaled to standardized form in all cases using the relationship $v_i' = (v_i - v_{mid})/(0.5*(v_{max} - v_{min}))$.

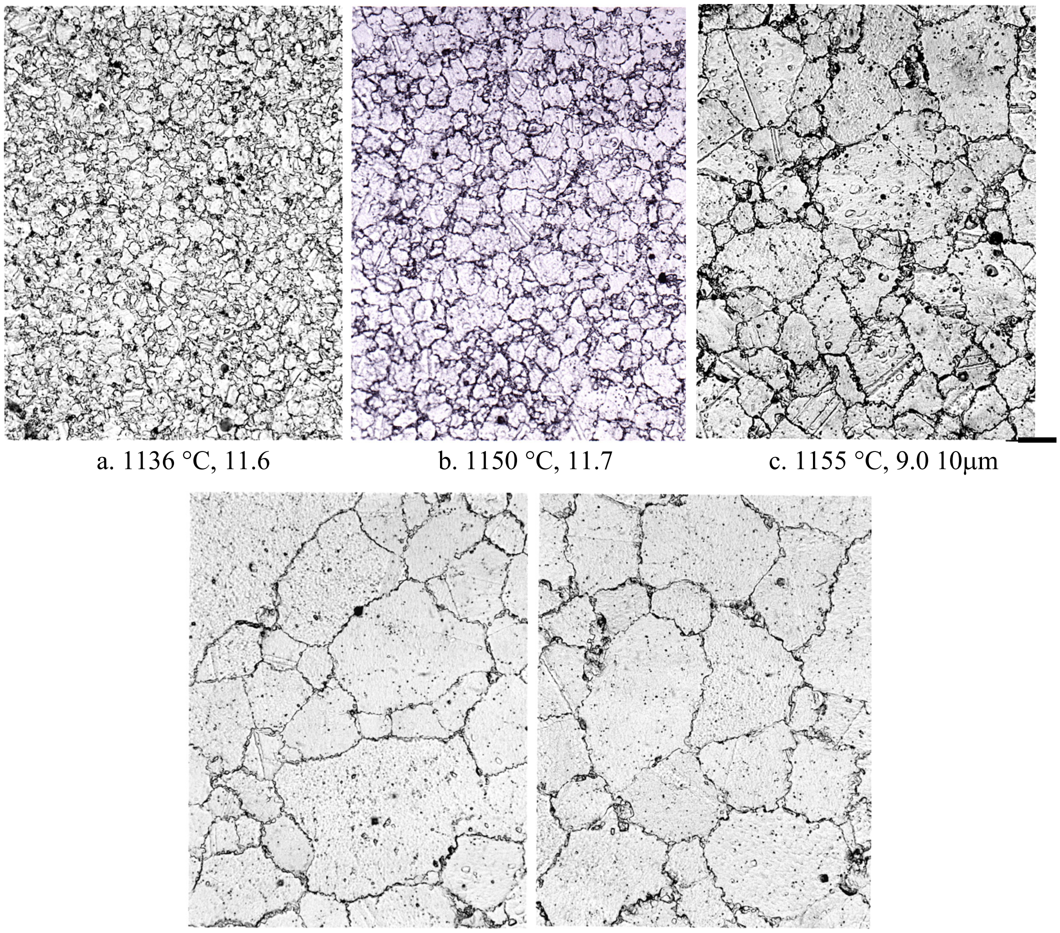
TABLE 1.—ACTUAL ALLOY COMPOSITIONS (WT%) AND GRAIN SIZE TRANSITION TEMPERATURES (T_g)

Alloy	Al	B	C	Co	Cr	Mo	Nb	Ta	Ti	W	Zr	Ni	T_g – C
ME3	3.41	0.024	0.050	20.56	12.96	3.71	0.88	2.28	3.64	2.06	0.047	50.38	1153
77	3.24	0.024	0.040	20.51	13.3	2.86	0.91	2.33	3.61	2.08	0.050	51.05	1154
78	3.42	0.027	0.035	20.44	13.16	2.83	0.90	2.42	3.63	2.98	0.050	50.11	1153
79	3.35	0.026	0.036	20.38	13.18	3.27	0.89	2.62	3.52	3.67	0.048	49.01	1153
80	3.41	0.028	0.038	20.50	13.17	2.87	0.89	2.12	3.58	4.10	0.048	49.25	1153
81	3.30	0.028	0.036	20.60	13.03	3.71	0.90	2.32	3.61	2.98	0.048	49.44	1153
NF3	----	------	------	------	------	----	-----	-----	-----	-----	------	------	1179
A	3.80	0.030	0.042	17.00	11.00	2.50	0.90	1.00	3.80	5.60	0.100	54.23	1176
B	3.90	0.030	0.045	19.20	11.20	2.60	0.90	1.00	3.80	5.80	0.100	51.43	1171
C	3.90	0.030	0.042	15.20	11.20	2.60	0.80	1.80	3.80	5.90	0.100	54.63	1186
D	3.90	0.030	0.034	15.30	11.20	2.60	0.01	1.80	3.80	5.70	0.100	55.53	1185
E	4.00	0.030	0.042	17.10	11.30	2.60	0.90	1.90	3.80	5.60	0.100	52.63	1186
F	3.90	0.030	0.045	15.70	11.20	2.60	1.70	1.00	3.80	5.80	0.100	54.13	1186
Alloy 10	3.90	0.030	0.036	15.20	11.00	2.60	0.80	1.00	3.80	5.60	0.100	55.93	1182

Results and Discussion

Comparison of Microstructure Versus Temperature

The typical grain microstructures are compared as a function of several temperatures for a typical alloy in figure 1. Grain size versus temperature is shown for all the alloys in figure 2. Grain size increased and undissolved γ′ phase content gradually decreased with increasing temperature. Grain size increased from about ASTM 11–12 (5 to 8 μm nominal diameter) to ASTM 7–8 (22 to 32 μm). Undissolved γ′



a. 1136 °C, 11.6 b. 1150 °C, 11.7 c. 1155 °C, 9.0 10μm

d. 1159 °C, 8.0 e. 1193 °C, 7.3

Figure 1.—Grain structure versus temperature, ASTM grain sizes for alloy 78.

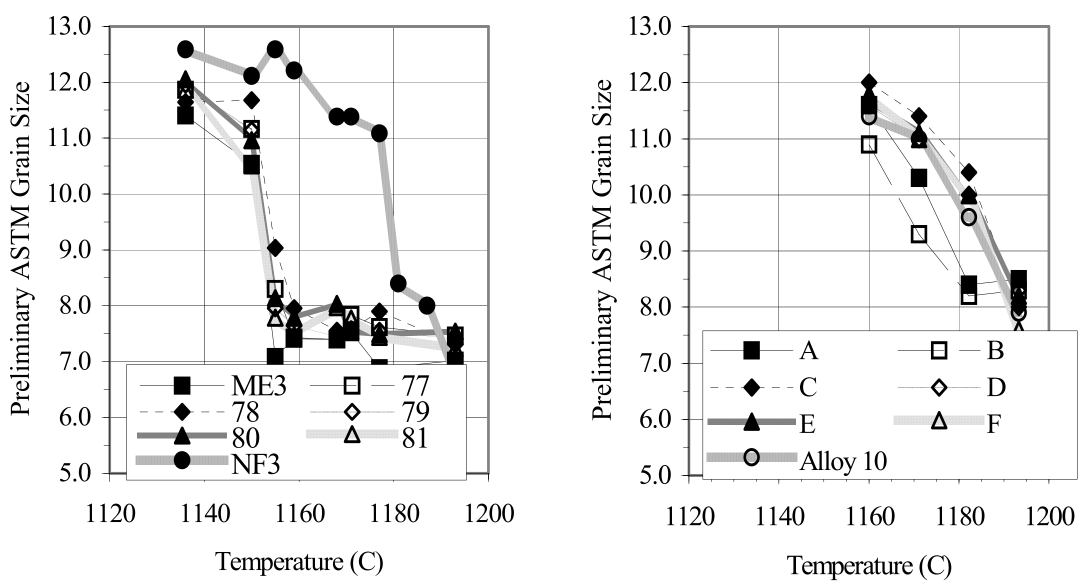
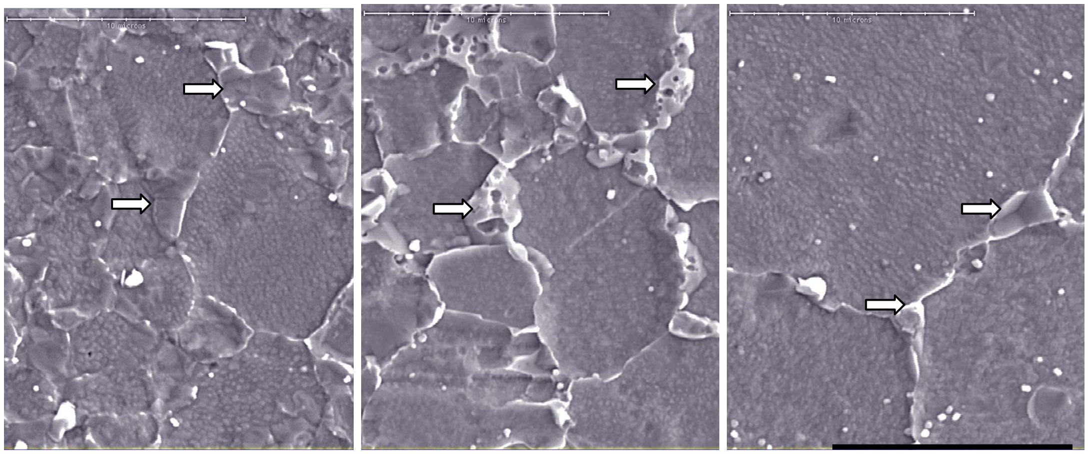
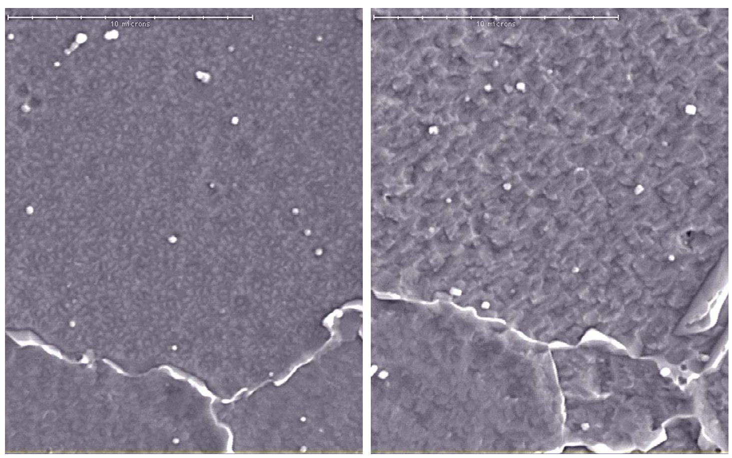


Figure 2.—ASTM grain size versus temperature for the experimental alloys.



a. 1136 °C b. 1150 °C c. 1155 °C 10μm



d. 1159 °C e. 1193 °C

Figure 3.—Grain boundary microstructure versus temperature for alloy 78, primary γ′ particles pinning grain boundaries indicated by arrows.

phase precipitate content was decreased to negligible levels in the latter coarse grain microstructures, figure 3. This can be attributed to the less constrained grain growth possible when large undissolved γ′ precipitates no longer pin the grain boundaries (ref. 3). The mean temperature at which the increase in grain size occurred was identified as the grain size transition temperature, T_g, for each alloy. This temperature was taken from the response curves as the temperature at a transitional grain size of ASTM 9.5 (13 μm). T_g could be estimated within ±3 °C for the ME3-based alloys and NF3, and within ±5 °C for the Alloy 10-based samples. For practical considerations, this T_g temperature is a most usable parameter for heat treatment design. Bearing in mind typical furnace tolerances of ±6 °C, supersolvus and subsolvus heat treatments can be specified at sufficiently lower and higher temperatures than this transition temperature, to insure the attainment of consistent grain sizes near ASTM 11–12 and ASTM 7–8, respectively. This temperature could also be used as a practical approximation of the γ′ phase solvus temperature. The grain size transition temperature generally occurred at a higher temperature for Alloy 10 alloys and NF3 than ME3 alloys. However, the grain size transition was more gradual for Alloy 10 alloys than for ME3 and NF3 alloys. The grain size transition temperatures are included versus alloy chemistry in table 1.

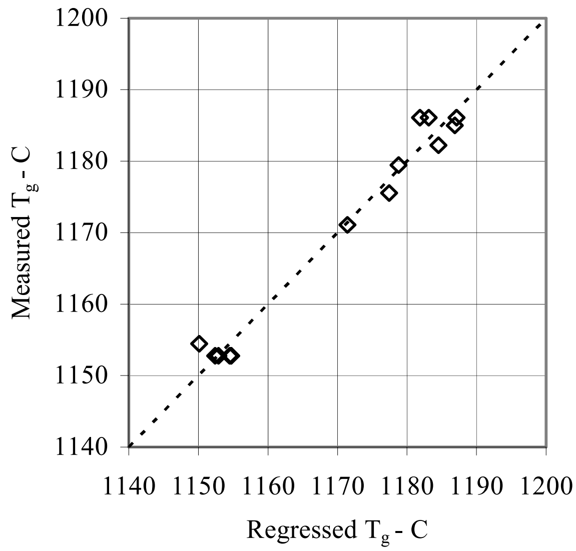


Figure 4.—Regressed versus measured T_g temperatures.

Regression of Grain Size Response

Forward and reverse stepwise linear regression was performed on the grain size transition temperatures versus alloy chemistry, using an F-to-enter of 4. The following regression equation was derived:

$$T_g = 1172.5 + 6.6\text{Al}' - 7.7\text{Co}' - 10.5\text{Cr}' + 3.6\text{Ta}'$$

where elements are expressed in wt% and Al′ = (Al – 3.62)/0.38; Co′ = (Co – 17.9)/2.7; Cr′ = (Cr – 11.5)/1.8; Ta′ = (Ta – 1.81)/0.81, and grain size transition temperature T_g is given in degrees C. This equation had an adjusted correlation coefficient R^2_{adj} = 0.97 and root mean square error of 2.6 °C, indicating acceptable predictive capability. A plot of regressed versus measured T_g temperatures is shown in figure 4.

The full statistical output is given in the appendix. The adjusted effects plots in the appendix show the effects of each of these variables on T_g temperature. For each variable, the T_g temperature has been adjusted to take out the effects of all other significant variables using the above equation. This allows clear inspection of the effect of each variable alone on T_g. These plots clearly show that increasing Al and Ta increased T_g, while increasing Co and Cr decreased T_g.

Summary and Conclusions

A series of experimental alloys based on ME3 and Alloy 10 were consolidated, extruded, and heat treated to determine the grain size-temperature responses. The findings can be summarized as follows:

(1) Grain size of disk alloys could be predictably controlled by proper selection of solution heat treatment temperatures.

(2) ME3-based alloys had generally sharper grain size transition curves, while Alloy 10-based compositions had more gradual transition curves. This could be related to a very sluggish dissolution rate of primary γ′ particles in alloys containing higher Al, Ti, and W.

(3) Alloy 10 compositions generally had higher grain size transition temperatures than ME3 compositions.

(4) Regression analysis indicated the grain size transition temperature was increased with increasing Al and Ta levels, and decreased with increasing Co and Cr levels.

Appendix

Least Squares Coefficients, Response TG, Model DESIGN__COPY

Term	Coeff.	Std. Error	T-value	Signif.
1 1	1172.454563	0.835768		
2 ~AL	6.628042	2.402410	2.76	0.0222
3 ~CO	-7.749442	1.826000	-4.24	0.0022
4 ~CR	-10.452189	1.580244	-6.61	0.0001
5 ~TA	3.599960	1.572661	2.29	0.0478

Term	Transformed Term
1 1	
2 ~AL	((AL-3.62)/3.8e-01)
3 ~CO	((CO-1.79e+01)/2.7)
4 ~CR	((CR-1.15e+01)/1.8)
5 ~TA	((TA-1.81)/8.1e-01)

No. cases = 14   R-sq. = 0.9791   RMS Error = 2.616
Resid. df = 9   R-sq-adj. = 0.9698   Cond. No. = 5.583
~ indicates factors are transformed.

1 STEP
2 Obey HIERARCHY
3 KEEP In
> 4 Display DATA
5 All SUBSETS
> 6 Show COEFFICIENT
7 HISTORY/PRESS
8 POOL Mixture
9 COLLINEARITY
10 RESPONSE/MODEL
11 NEXT
12 MAIN

Least Squares Summary ANOVA, Response TG Model DESIGN__COPY

Source	df	Sum Sq.	Mean Sq.	F-Ratio	Signif.
1 Total(Corr.)	13	2948.857			
2 Regression	4	2887.288	721.822	105.50	0.0000
3 Residual	9	61.569	6.841		

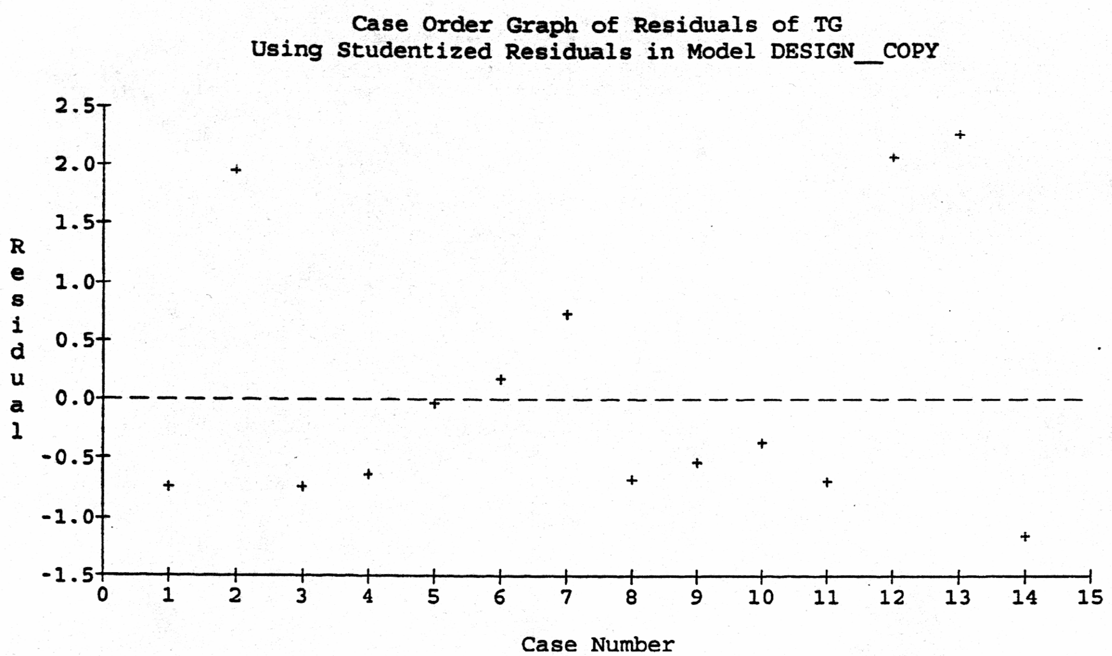
R-sq. = 0.9791
R-sq-adj. = 0.9698
Model obeys hierarchy. The sum of squares for each term is computed assuming higher order terms are first removed.

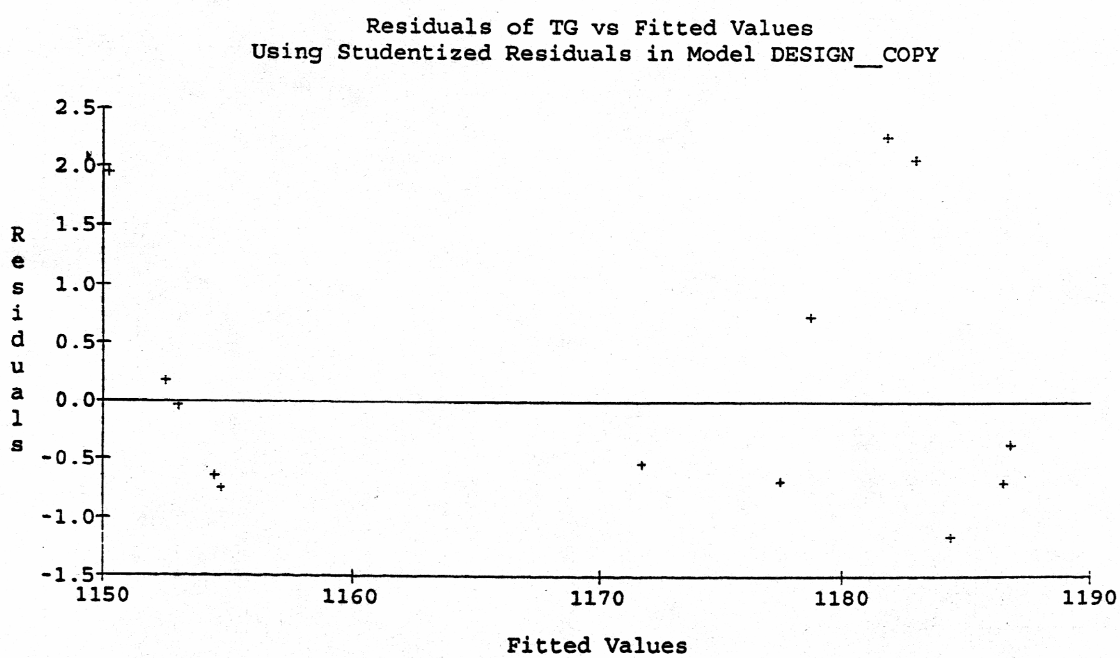
Least Squares Components ANOVA, Response TG Model DESIGN__COP

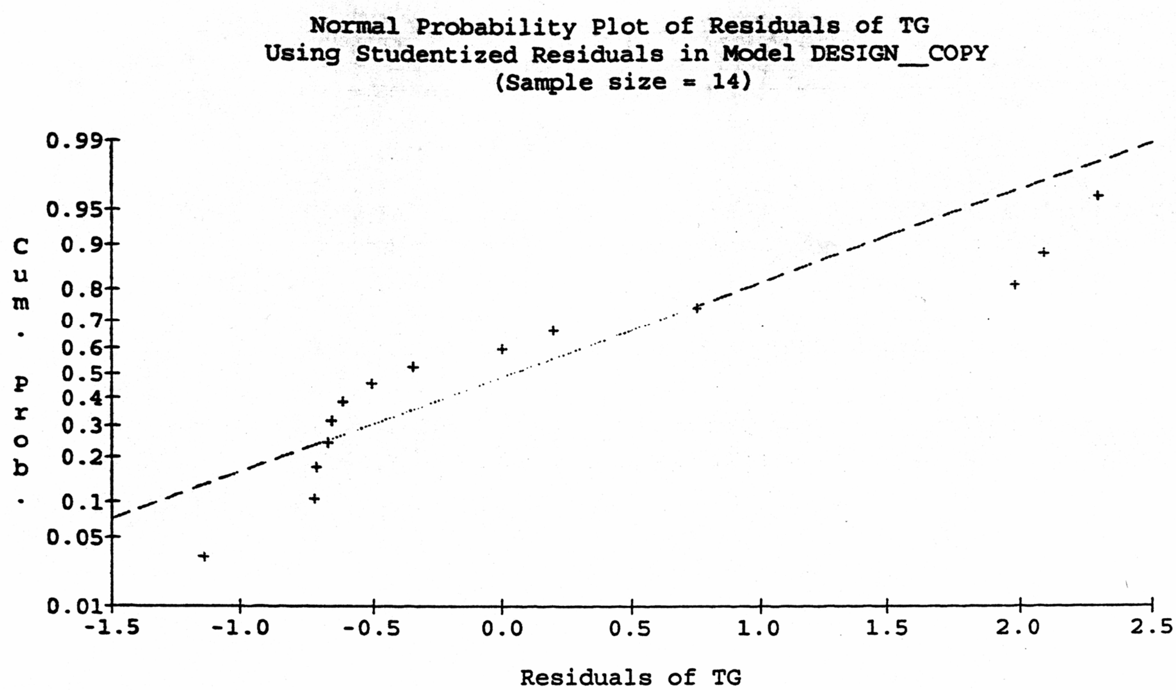
Source	df	Sum Sq.	Mean Sq.	F-Ratio	Signif.
1 Constant	1	19141207			
2 ~AL	1	52.071	52.071	7.61	0.0222
3 ~CO	1	123.213	123.213	18.01	0.0022
4 ~CR	1	299.285	299.285	43.75	0.0001
5 ~TA	1	35.846	35.846	5.24	0.0479
6 Residual	9	61.569	6.841		

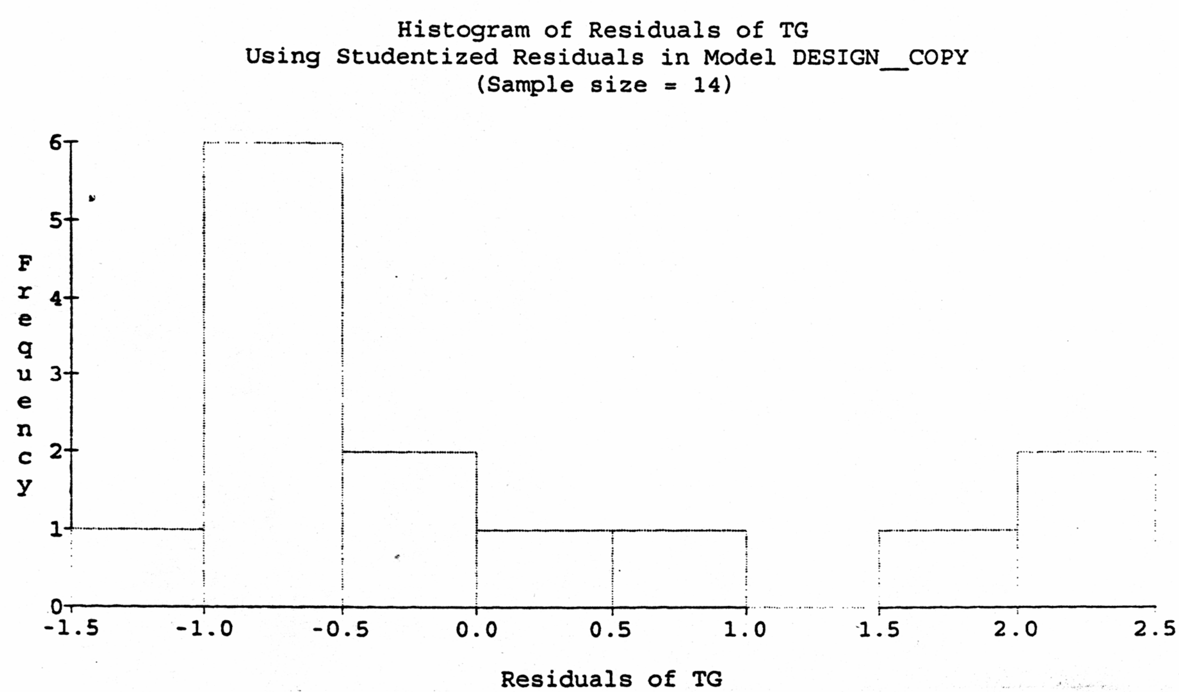
~ indicates factors   R-sq. = 0.9791
are transformed.   R-sq-adj. = 0.9698
Default sum of squares.
Model obeys hierarchy. The sum of squares for each term is computed assuming higher order terms are first removed.

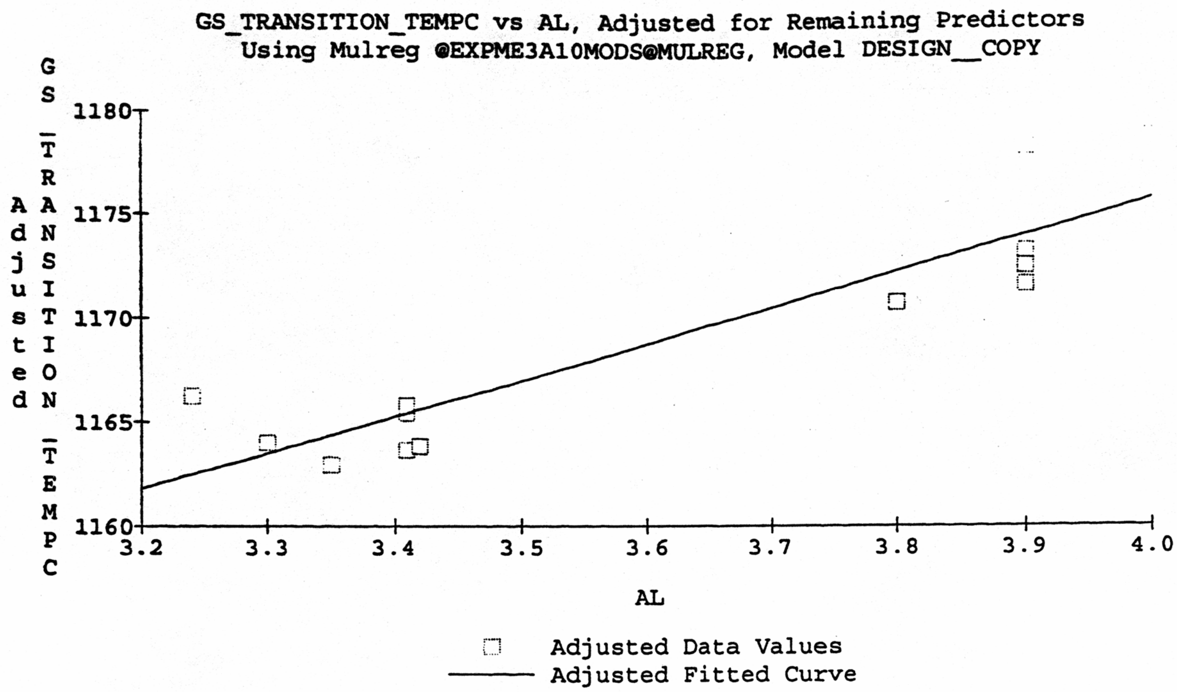
1 SUMMARY Anova
> 2 COMPONENTS Anova
3 VARIANCES
4 FIXED Effects
5 RANDOM Effects
6 MIXTURE Pooling
7 FULL Factorial
8 INTERPRETATION
9 RESPONSE/MODEL
10 OPTIONS
11 NEXT
12 MAIN


Case Order Graph of Residuals of TG
Using Studentized Residuals in Model DESIGN__COPY
Residual
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
-1.5
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15
Case Number

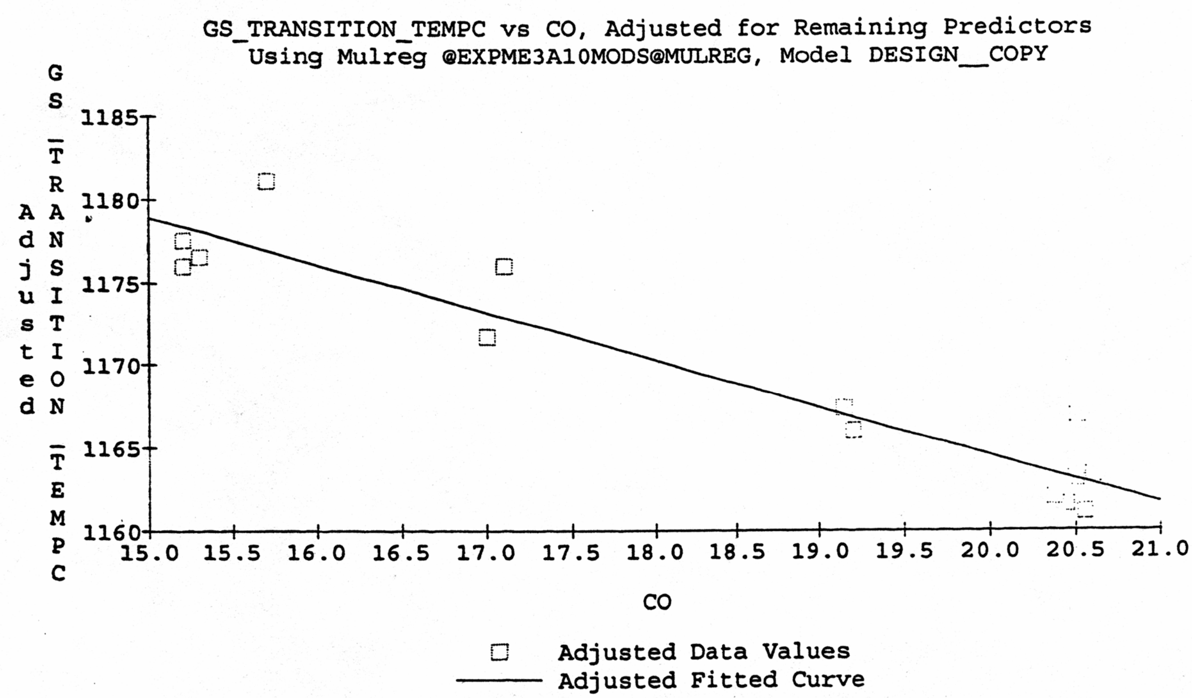

Residuals of TG vs Fitted Values
Using Studentized Residuals in Model DESIGN__COPY
Residuals
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
-1.5
1150 1160 1170 1180 1190
Fitted Values


Normal Probability Plot of Residuals of TG
Using Studentized Residuals in Model DESIGN__COPY
(Sample size = 14)
Cum. Prob.
0.99
0.95
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0.05
0.01
-1.5
-1.0
-0.5
0.0
0.5
1.0
1.5
2.0
2.5
Residuals of TG

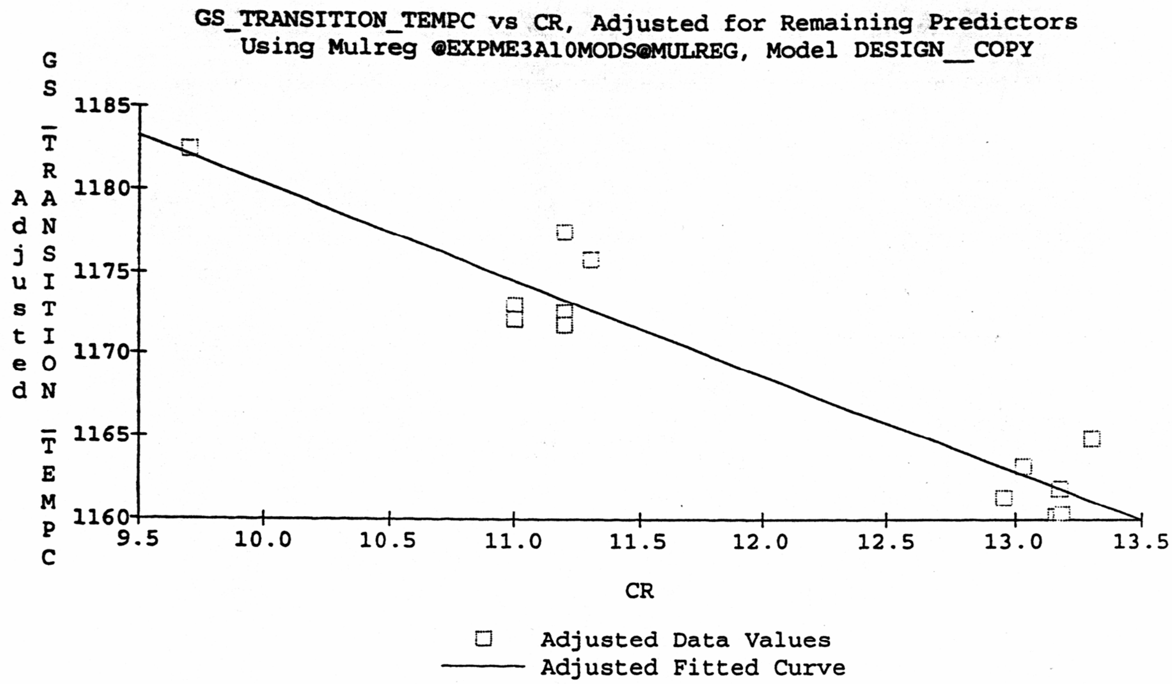

Histogram of Residuals of TG
Using Studentized Residuals in Model DESIGN__COPY
(Sample size = 14)
Frequency
6
5
4
3
2
1
0
-1.5
-1.0
-0.5
0.0
0.5
1.0
1.5
2.0
2.5
Residuals of TG

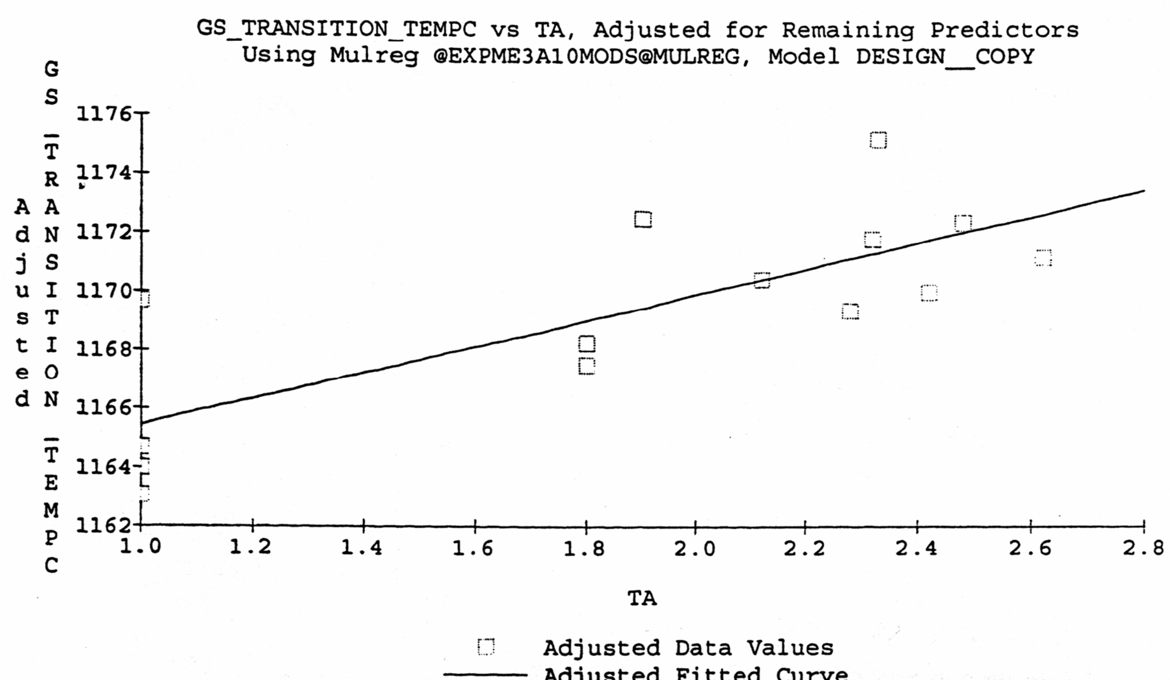


GS_TRANSITION_TEMPC vs AL, Adjusted for Remaining Predictors
Using Mulreg @EXPME3A10MODS@MULREG, Model DESIGN__COPY
Adjusted GS_TRANSITION_TEMPC
1180
1175
1170
1165
1160
3.2
3.3
3.4
3.5
3.6
3.7
3.8
3.9
4.0
AL
Adjusted Data Values
Adjusted Fitted Curve

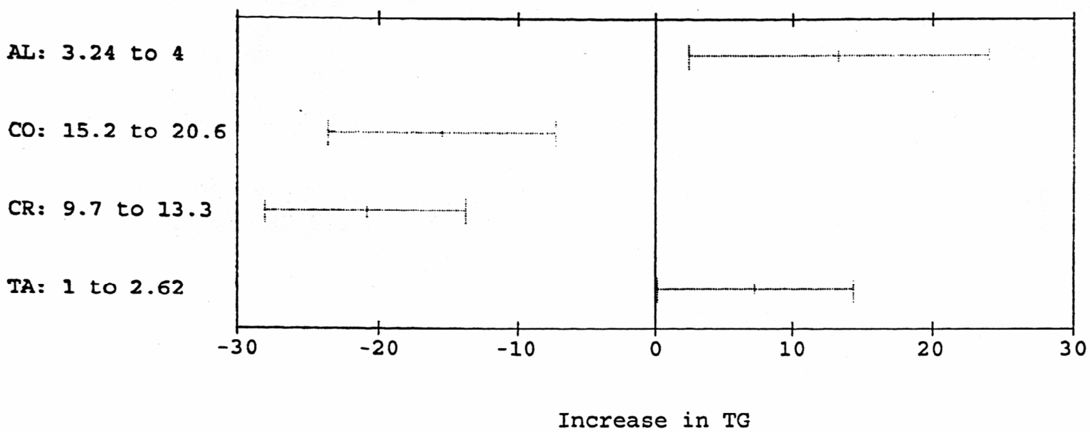


GS_TRANSITION_TEMPC vs CO, Adjusted for Remaining Predictors
Using Mulreg @EXPME3A10MODS@MULREG, Model DESIGN__COPY
Adjusted GS_TRANSITION_TEMPC
1185
1180
1175
1170
1165
1160
15.0
15.5
16.0
16.5
17.0
17.5
18.0
18.5
19.0
19.5
20.0
20.5
21.0
CO
Adjusted Data Values
Adjusted Fitted Curve


GS_TRANSITION_TEMPC vs CR, Adjusted for Remaining Predictors
Using Mulreg @EXPME3A10MODS@MULREG, Model DESIGN__COPY
Adjusted GS_TRANSITION_TEMPC
1185
1180
1175
1170
1165
1160
9.5
10.0
10.5
11.0
11.5
12.0
12.5
13.0
13.5
CR
Adjusted Data Values
Adjusted Fitted Curve


GS_TRANSITION_TEMPC vs TA, Adjusted for Remaining Predictors
Using Mulreg @EXPME3A10MODS@MULREG, Model DESIGN__COPY
Adjusted GS_TRANSITION_TEMPC
1176
1174
1172
1170
1168
1166
1164
1162
1.0
1.2
1.4
1.6
1.8
2.0
2.2
2.4
2.6
2.8
TA
Adjusted Data Values
Adjusted Fitted Curve

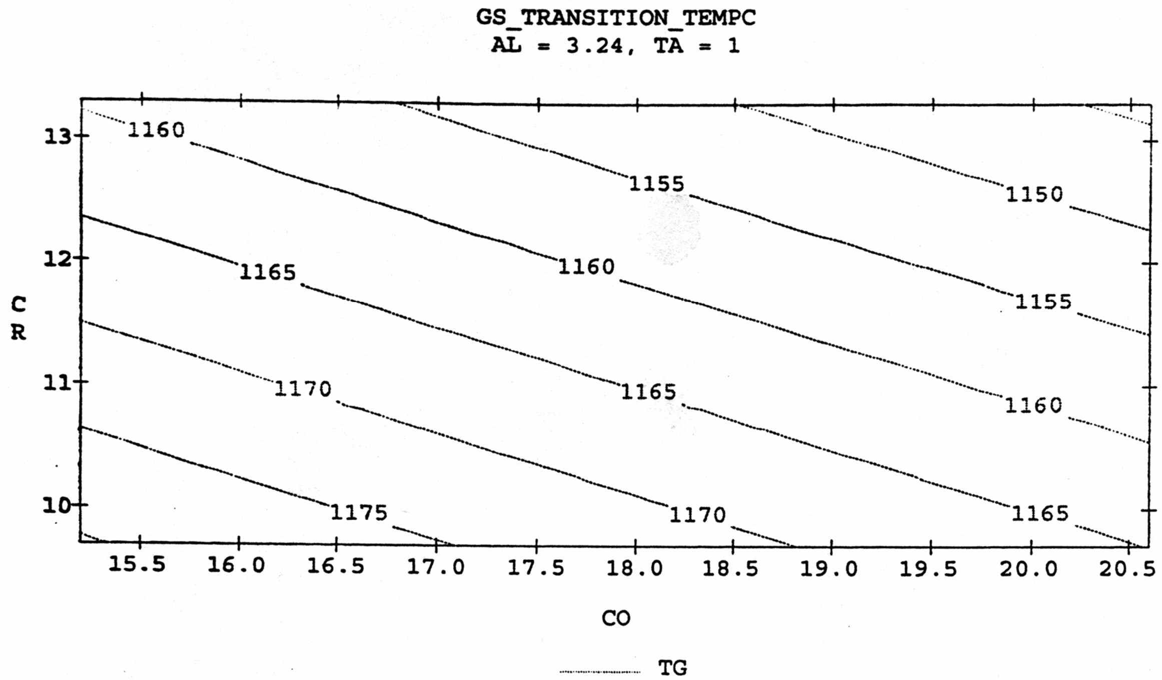
Mulreg @EXPME3A10MODS@MULREG, Model DESIGN__COPY
Main Effects on Response GS_TRANSITION_TEMPC
(with 95% Confidence Intervals)

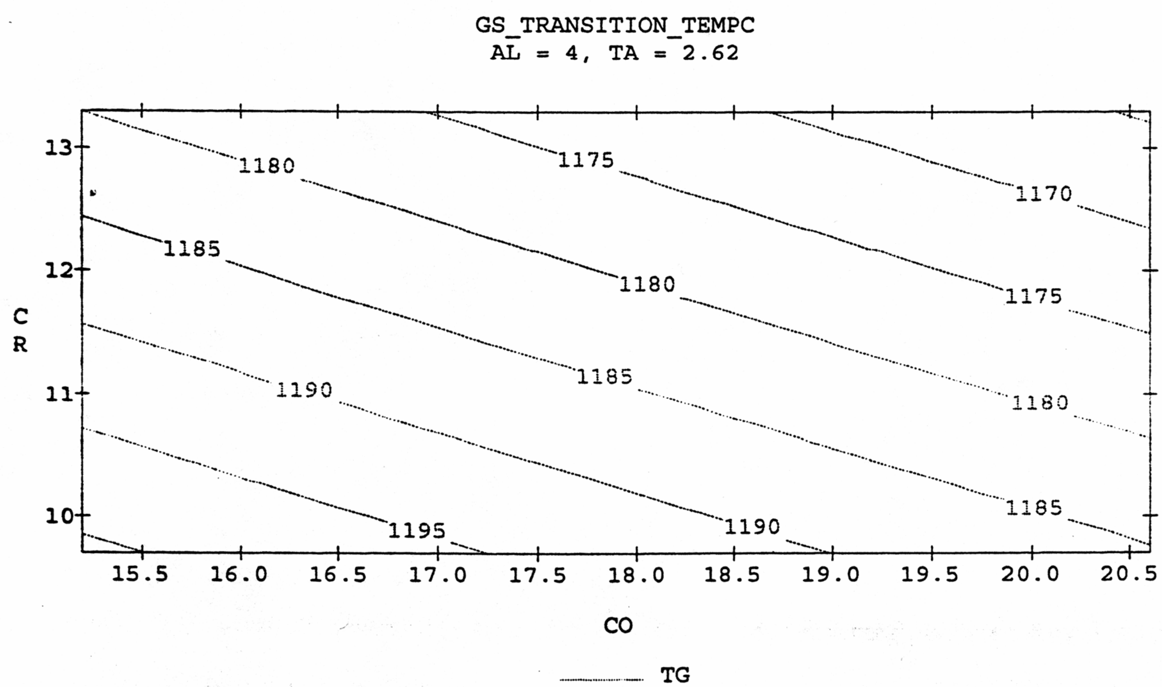


Factor, Response or Formula	Range	Initial Setting	Optimal Value
1 Factors			
2 AL	3.24 to 4	3.62	3.2401
3 CO	15.2 to 20.6	17.9	20.6
4 CR	9.7 to 13.3	11.5	13.299
5 TA	1 to 2.62	1.81	1.1157
6			
7 Responses			
8 GS_TRANSITION_T	MIN		1144.5

1 SELECT
2 MIN/MAX
3 FACTOR Ranges
4 CONSTRAINTS
5 INITIAL Settings
6 TOLERANCE
7 STEP Limit
8 PERFORM
> 9 STORE
10 RECALL
11 NEXT
12 MAIN

Converged to a tolerance of 0.0033 after 118 steps.


GS_TRANSITION_TEMPC
AL = 3.24, TA = 1
1160
1155
1150
1165
1160
1155
1170
1165
1160
1175
1170
1165
13
12
11
10
C R
15.5
16.0
16.5
17.0
17.5
18.0
18.5
19.0
19.5
20.0
20.5
CO
TG


GS_TRANSITION_TEMPC
AL = 4, TA = 2.62
1180
1175
1170
1185
1180
1175
1190
1185
1180
1195
1190
1185
13
12
11
10
C R
15.5
16.0
16.5
17.0
17.5
18.0
18.5
19.0
19.5
20.0
20.5
CO
TG

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